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PROVISIONAL APPLICATION FOR PATENT COVER SHEET
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LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)		
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TITLE OF THE INVENTION (280 characters max)					
POTTING DEVICES AND METHODS OF MAKING					
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<input type="checkbox"/> Applicant(s) claim(s) small entity status. See 37 C.F.R. § 1.27. <input checked="" type="checkbox"/> A check or money order is enclosed to cover the Provisional filing fees <input checked="" type="checkbox"/> The Commissioner is hereby authorized to additional fees to Deposit Account Number: 50-0436				PROVISIONAL FILING FEE AMOUNT (\$)	\$160.00

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☐ Yes, the name of the U.S. Government agency and the Government contract number are: _____

☐ Additional inventors are being named on separately numbered sheets attached hereto.

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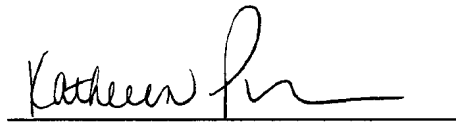
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APPLICANT: JOSEPH SMITH
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DOCUMENTS ENCLOSED:

1. New Provisional Application Cover Sheet (1 sheet);
2. Provisional Application (33 pages);
3. Formal Figures (6 sheets)
4. Fee Transmittal and check in the amount of \$160.00; and
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POTTED DEVICES AND METHODS OF MAKING

BACKGROUND

[0001] Hollow fibers and thin walled hollow tubes are commonly used in mass transfer, heat exchange, and cross flow particle filtration devices. In these applications the hollow tubes or porous fibers provide a high surface to volume ratio which permits a greater transfer of heat and mass in a smaller volume than a device made with flat sheet materials of similar composition.

[0002] A hollow fiber or a hollow tube includes an outer diameter and surface, an inner diameter and surface, and a porous or non-porous material between the first and second surfaces or sides of the tube or fiber. The inner diameter defines the hollow portion of the fiber or tube and is used to carry one of the fluids. For what is termed tube side contacting, a first fluid phase flows through the hollow portion, sometimes called the lumen, and is maintained separate from a second fluid phase, which surrounds the tube or fiber. In shell side contacting, the first fluid phase surrounds the outer diameter and surface of the tube or fibers and the second fluid phase flows through the lumen. In an exchange apparatus, packing density relates to the number of useful hollow fiber or hollow tubes that can be potted in the apparatus.

[0003] Examples of applications in semiconductor manufacturing where heating of a liquid is required include sulfuric acid and hydrogen peroxide photoresist strip solutions, hot phosphoric acid for silicon nitride and aluminum metal etching, ammonium hydroxide and hydrogen peroxide SC1 cleaning solutions, hydrochloric acid and hydrogen peroxide SC2

cleaning solutions, hot deionized water rinses, and heated organic amine based photoresist strippers

[0004] Previously, devices were potted by preparing a shell or housing by sintering a thin layer of the potting material to the inside diameter surface of the shell. The device assembly, including media, was potted by surrounding the assembly with resin, and raising the temperature in a controlled environment to melt the resin, thus fill the space around the media at one end of the device. The resin would flow sufficiently into the shell to encapsulate the media and adhere to the sidewall of the shell that had been sintered with the like material in an earlier step. Other potting methods previously used include: potting the device, hollow tubes, outside of the shell, machining the potted area to create a bonding surface and thermally bonding the potted material to a housing; potting the device outside of the shell, machining the potted area to create a mating flange and sealing surface, which would be mechanically joined to the shell using an o-ring or other contact sealing method- these parts could be held together by a snap ring, threaded fasteners and or a secondary bonding operation. Additional methods include inserting pins in each fiber, potting the assembly and removing the pins after potting. Some devices are made by filling the lumen i.d. with a binder and extracting out the binder after potting and machining.

SUMMARY

[0005] Embodiments of the present invention include a housing or shell for a thermoplastic potted hollow tube or membrane device which includes grooves or channels on the inner a surface of the housing. The thermoplastic potting material fills at least a portion of the grooves in the housing surface and bonds to a portion of them to form a unitary end structure. The grooves permit potted devices to be used at higher temperatures and pressures while maintaining the fluid integrity of the unitary end structure. Devices of the present invention and

methods for making them include any device that requires potting the working media into a housing meant for containment of the process fluid including but not limited to fully bonded membrane contactors, gas contactors, ozone contactors, degassers, heat exchangers, gas scrubbers, hollow fiber filters, and combinations of these.

[0006] The present invention improves the strength of such potted devices by creating a mechanical interlock and or a fused bond between the potting material and the channels or grooves in the housing shell. The interlock and or bond serves as a molded in sealing surface, that has mechanical strength, preventing separation of the mating parts. The grooves, and their additional surface area, some of which is not parallel to the housing walls, result in fusion and adhesion of the potting resin to at least a portion of the surfaces of the groove. This adds a shear component to the radial force created by thermal or pressure expansion of the housing shell. This shear component greatly improves the strength of the device.

[0007] One embodiment of the present invention is an exchange device that includes a thermoplastic housing having one or more thermoplastic hollow tubes or porous fibers or a combination of these fluidly sealed to at least one end of the housing by a thermoplastic resin. The thermoplastic resin fluidly sealed (by fusion) to an end portion of said housing by one or more grooves in said housing; said grooves and resin forming a unitary end structure wherein the resin and housing with grooves fuse at a portion of the housing to form said unitary end structure. The exchange device may have a sintered thermoplastic coating on the inside of the housing including the surfaces of the grooves. The exchange device housing may include fluid fittings on the shell side and tube side of the device. The exchange device preferably includes the a thermoplastic and preferably the thermoplastic is MFA. The exchange device may include but is not limited to potted hollow tube which are porous hollow fibers, skinned hollow fibers,

thermoplastic tubes, or combinations of these. Preferably the ends of the potted hollow tubes are open. The mechanical interlock and fusion of the potting resin with the housing grooves serves to entrain the shell and potted area together, particularly when stressed due to temperature or pressure. The shell is prevented from expanding more than the potted material, keeping the assembly (housing, potting and hollow tubes and or fibers) integral.

[0008] Another embodiment of the present invention is an exchange device that includes a thermoplastic housing having one or more fluidly sealed hollow tubes potted in a thermoplastic resin where the thermoplastic resin occupies a volume of one or more grooves on an interior surface of the housing. The grooves and resin form a unitary end structure wherein the thermoplastic resin and housing fuse at a portion of the groove to form said unitary end seal. The housing inner surface and grooves may be coated with a sintered thermoplastic material to which the potting resin may fuse.

[0009] Another embodiment of the present invention is an apparatus for exchanging energy or mass with a process fluid. The exchange device may have a process fluid inlet and outlet connected to the housing for receiving and delivering a process fluid into a re-circulation loop or to a dispense tool. The exchange device can have an exchange fluid inlet and outlet fittings for flow of an exchange fluid; the exchange fluid separated from the treated fluid by the material in the walls of the hollow tubes. The exchange fluid exchanges or transfers mass and or energy to or from the process fluid through the hollow tube walls. The exchange device of the present invention may be used in an apparatus that optionally includes a re-circulating pump in fluid communication with the process fluid inlet on the exchange device and a tank for holding an article to be treated by the process fluid. The exchange device may be used as part of a dispense system or a re-circulating fluid flow circuit. The apparatus may also further include a

particle filter. The apparatus may exchange mass or energy with organic liquids or aqueous fluids including ultra high purity water. Preferably the substrate or article to be treated by the process fluid includes but is not limited to metals such as copper and aluminum, semiconductors including arsenic or silicon, and ceramics including aluminum, barium, and strontium.

[0010] Another embodiment of the present invention is an exchange device that includes a thermoplastic housing having one or more fluidly sealed hollow tubes potted in a thermoplastic resin. The resin occupies a volume of one or more grooves on an interior surface of the housing. Preferably the grooves and resin form a unitary end structure wherein the resin and housing fuse at a portion of the groove or a coating thereon to form the unitary end seal. The device may be used to heat a fluid while maintaining the fluid integrity of bond between the housing and the potting resin at a temperature of at least 50 °C, preferably at least 130 °C, and more preferably above 150 °C. Preferably the exchange device maintains its fluid integrity at these temperatures when the pressure of the fluid is at least 10 psig, preferably at least 50 psig, and more preferably 70 psig or greater.

[0011] Another embodiment of the present invention is a method of making an exchange device that includes flowing a thermoplastic material into a housing, the housing having grooves on an inner surface of the tube and one more hollow tubes within the housing. The method further includes forming a fluid tight seal between said thermoplastic material and the hollow tubes and a fluid tight seal between said thermoplastic potting resin and the housing. Preferably the thermoplastic potting resin occupies at least a portion of the grooves in the housing and even more preferably the resin fuses with a portion of the groove or a sintered thermoplastic material

coating the housing and groove surfaces. Preferably the housing has a coating of a sintered thermoplastic material on one or more of the housing surfaces.

[0012] Another embodiment of the present invention is a method of treating a fluid that includes flowing a process fluid to be treated on a first side of at least one hollow tube having two sides and a thermoplastic wall interposed between them, the hollow tubes potted in a fluid tight manner within a thermoplastic material. The thermoplastic potting material is bonded to a housing for the device that has one or more grooves on the inside surface of the housing. At least a portion of the housing grooves are filled with and bonded to the thermoplastic material to form a fluid tight seal between the thermoplastic material, the hollow tube and the housing. The method includes exchanging energy, mass, or a combination of these with the process fluid to be treated by flowing an exchange fluid on a second side of the hollow tubes. The energy, mass, or a combination of these is transferred to or from the process fluid to the exchange fluid through the hollow tube wall. The hollow tubes may be non-porous, porous, or a combination of these.

DESCRIPTION OF THE DRAWINGS

[0013] In part, other aspects, features, benefits and advantages of the embodiments of the present invention will be apparent with regard to the following description, appended claims and accompanying drawings where:

[0014] FIG. 1 is an illustration of an embodiment of a device for heat and or mass transfer of the present invention illustrating a housing with endcaps which has hollow tubes or hollow fiber, or a combination of these fluidly sealed by potting them into a thermoplastic resin, the resin bonded into the housing to form a unitary end structure at each end of the housing, the potting material, which is preferably a thermoplastic is shown filling at least a portion of a groove or channel formed in the housing;

[0015] FIG. 2 is an illustration of non-limiting groove or channel shape designs for adding mechanical advantage to the interface between the shell and potting; the channels may be coated with a sintered thermoplastic material for bonding and may have various shapes including but not limited to rectangular grooves (B), wedge shaped groove (A), and combinations of these or others, the width of the grooves, their depth, their spacing from one another, and their spacing from the end of the housing may be varied without limitation;

[0016] FIG. 3 is a schematic illustration of a housing including channels with thermoplastic resin filling at least a portion of the channels and preferably fusing to a portion of the housing; hollow tubes, porous hollow fibers, or a combination of these are shown potted into the resin to form a fluid tight seal and forming a unitary end structure with the housing walls and grooves; the housing walls and grooves may have fused to their surface sintered thermoplastic material.

[0017] FIG. 4 is an illustration of a heat exchanger of the present invention, with grooves in the housing and hollow tubes potted to form a fluid tight seal in a thermoplastic resin which forms a unitary end structure with the housing walls and grooves, used as part of an apparatus for conditioning the temperature of a process fluid used to clean or chemically modify substrates in a bath or a substrate holder such as a single wafer cleaning tool (not shown);

[0018] FIG. 5 is an illustration of a heat exchanger of the present invention, with grooves in the housing and hollow tubes potted to form a fluid tight seal in a thermoplastic resin which forms a unitary end structure with the housing walls and grooves in the housing; the device is used as part of an apparatus for cooling a heated process fluid prior to discharge.

[0019] FIG. 6 is an illustration of an energy exchanger of the present invention with grooves in the housing and hollow tubes or hollow fibers potted to form a fluid tight seal in a

thermoplastic resin which forms a unitary end structure with the housing walls and grooves in the housing; the device is used as part of an apparatus for cooling a heated process fluid prior to discharge on to a single substrate which may be rotating or stationary. In this non-limiting illustration, fluid from a fluid source which may be for cleaning the substrate or for coating it is filtered with an optional particle filter and energy from the fluid is added or removed by the heat exchanger as it passes in a counter-current manner through the exchanger. The heat exchanger exchanging energy with the fluid through the hollow tube walls with the exchange fluid. Temperature, pressure and flow controllers may be used to control the amount of fluid dispensed onto the substrate. The energy exchanged process, coating, or cleaning fluid can be delivered by a pump to a substrate which may be stationary or rotating. The location of the particle filter, valves, flow controller and pump (or the fluid source may be pressurized) may be changed without limitation.

DETAILED DESCRIPTION

[0020] Before the present compositions and methods are described, it is to be understood that this invention is not limited to the particular molecules, compositions, methodologies or protocols described, as these may vary. It is also to be understood that the terminology used in the description is for the purpose of describing the particular versions or embodiments only, and is not intended to limit the scope of the present invention which will be limited only by the appended claims.

[0021] It must also be noted that as used herein and in the appended claims, the singular forms “a”, “an”, and “the” include plural reference unless the context clearly dictates otherwise. Thus, for example, reference to a “hollow tube” is a reference to one or more hollow tubes and equivalents thereof known to those skilled in the art, and so forth. Unless defined otherwise, all

technical and scientific terms used herein have the same meanings as commonly understood by one of ordinary skill in the art. Although any methods and materials similar or equivalent to those described herein can be used in the practice or testing of embodiments of the present invention, the preferred methods, devices, and materials are now described. All publications mentioned herein are incorporated by reference. Nothing herein is to be construed as an admission that the invention is not entitled to antedate such disclosure by virtue of prior invention.

[0022] Incorporated herein by reference in their entirety are U.S. Pat. No. 6,582,496; United States provisional application serial No. 60/326,234 filed October 1, 2001 entitled Fluid Exchange Device, and U.S. provisional patent application No.60/326,357, filed October 1, 2001 under Applicants' reference number 200100293 (formerly MYKP-621).

[0023] A fluid-fluid phase contactor or energy exchange device of the present invention may be made from perfluorinated thermoplastic polymers for contacting a first fluid with a second fluid. The contactor or exchange device includes a bundle of a plurality of perfluorinated thermoplastic hollow fiber membranes or hollow tubes, which may be porous or non-porous, and have a first end and a second end. The membranes and tubes have an outer surface and an inner surface. For membranes the inner surface comprises a lumen, the membranes and hollow tubes may be selected from the group consisting of hollow tubes, hollow fiber membranes having a porous skinned inner surface, a porous outer surface, and a porous support structure between, hollow fiber membranes having a non-porous skinned inner surface, a porous outer surface, and a porous support structure between, hollow fiber membranes having a porous skinned outer surface, a porous inner surface, and a porous support structure between, and hollow fiber membranes having a non-porous skinned outer surface, a porous inner surface, and a porous

support structure between. Each end of the bundle of tubes or membranes can be potted with a liquid tight perfluorinated thermoplastic seal forming a unitary end structure with a surrounding perfluorinated thermoplastic housing wherein the fiber ends are open to fluid flow. The housing has an inner wall with channels or grooves, a portion of which are filled with the thermoplastic potting resin, and an outer wall wherein the inner wall defines a fluid volume between the inner wall and the hollow tubes or the hollow fiber membranes. The housing preferably has a first fluid inlet to supply a first fluid to said first end of the bundle to be contacted with a second fluid and a first fluid outlet connection to remove said contacted first fluid from said second end. The housing preferably also has a second fluid inlet connection to supply a second fluid to be contacted with said first fluid through the walls of the hollow fibers or tubes. The second fluid occupies the volume formed between the inner wall of the housing and the hollow fiber membranes or tubes. The housing preferably includes an second outlet connection to remove said contacted second fluid.

[0024] A method of making a fluid-fluid phase contactor substantially made from perfluorinated thermoplastic polymers for contacting a first fluid with a second fluid may include forming a bundle of a plurality of perfluorinated thermoplastic hollow fiber membranes, hollow tubes, or a combination thereof having a first end and a second end. The membranes or tubes having an outer surface and an inner surface, said inner membrane surface comprising a lumen. The membranes or hollow tubes used in the device may be selected from the group consisting of hollow tubes, hollow fiber membranes having a porous skinned inner surface, a porous outer surface, and a porous support structure between, hollow fiber membranes having a non-porous skinned inner surface, a porous outer surface, and a porous support structure between, hollow fiber membranes having a porous skinned outer surface, a porous inner surface, and a porous

support structure between, and hollow fiber membranes having a non-porous skinned outer surface, a porous inner surface, and a porous support structure between. The hollow tubes or hollow membranes are positioned and surrounded by a perfluorinated thermoplastic housing having an inner wall with channels or grooves and an outer wall to form a fluid flow volume between the inner wall and the hollow fiber membranes. The hollow tubes and or fibers are potted at each end of the bundle positioned in the housing using a perfluorinated thermoplastic to form a liquid tight perfluorinated thermoplastic seal with the ends of the tubes or fibers and to form a unitary end structure with the surrounding perfluorinated thermoplastic housing including filling a portion of the channels or grooves with the thermoplastic. Preferably after potting the ends of the tubes are opened at both ends of the housing to provide fluid flow through the hollow fiber lumens or hollow tubes. Preferably the housing is provided with a first fluid inlet to supply a first fluid to the first end of the tubes or fibers to be contacted with a second fluid and a first fluid outlet connection to remove the contacted first fluid from said second end of the potted fibers or tubes. Preferably the housing is also provided with a second fluid inlet connection to supply a second fluid to be contacted with said first fluid to the volume formed between the inner wall of the housing and the hollow fiber membranes, and an second outlet connection to remove the contacted second fluid.

[0025] Potting and bonding of the tube cords into the housing can be done in a single step. The preferred thermoplastic resin potting material is Hyflon® MFA 940 AX resin, available from Ausimont USA Inc. Thorofare, NJ. The method comprises vertically placing a portion of a bundle of the annealed and twisted hollow tube cord lengths with at least one closed end into a pot lifted off the bottom surface of the pot by approximately 1/8 – 1/4 inch. Thermoplastic resin in pellet form is placed around the outside of the shell of the device, and the

pot is heated to 275 – 277 °C for hollow fiber device, 280-285 °C for thin wall tube devices. The resin melts and flows into the shell and between the lumens by head pressure and capillary action. An alternative method is to make a temporary recess made in a pool of molten thermoplastic polymer held in a container. The hollow tubes are held in a defined vertical position, maintaining the thermoplastic polymer in a molten state so that it flows into the temporary recess, around the hollow tubes and vertical up the fibers, completely filling the interstitial spaces between fibers with the thermoplastic polymer. A temporary recess is a recess that remains as a recess in the molten potting material for a time sufficient to position and fix the bundle of hollow tubes in place and then will be filled by the molten thermoplastic. The temporary nature of the recess can be controlled by the temperature at which the potting material is held, the temperature at which the potting material is held during hollow tube bundle placement, and the physical properties of the potting material. The end of the hollow tube can be closed by sealing, plugging, or in a preferred embodiment, by being formed in a loop.

[0026] For potting hollow tubes, where fusion of the tubes or fibers into the potting resins is desired, and forming a unitary end structure of the potting resin with the housing and channels or grooves in its surface, the process may involve placing the assembly in a heating cup and heating the potting resins in the heating cup with an external heating block or other heat source at a temperature in the range of from about 265 C to around 285 C, with a preferred range of from about 270 C to around 280 C, until the melt turns clear and is free of trapped bubbles. The resin flows up and between the fibers and in the shell until the height is equivalent or nearly equivalent on the inside of the shell as the outside of the shell. A secondary potting method rod is inserted into the melt to create a recess or cavity. The housing with the grooves or channels and the hollow tube bundle are then inserted into the cavity. It is important to note that at this

point neither the hollow tube bundle nor the housing touches the potting resin. The melted resin will flow by gravity to fill the voids and channels in the housing over time to pot the hollow tubes and bond to the housing simultaneously. After cooling the potting process may be repeated for the second end of the device. After the potted ends are cooled, they are then cut and the lumen of the hollow tubes exposed. The potted surfaces are then polished further using a heat gun to melt away any smeared or rough potted surfaces. For module with a large number of hollow tubes, such as 2000 or more, it is possible that the module may have potting defects which can be repaired using a clean soldering iron to fuse and close the damaged areas, or by melting new resin into the defect with the aid of a soldering iron.

[0027] Potting and bonding of hollow fibers to the thermoplastic resin, the housing, and grooves may be done in a single step. The method comprises vertically placing a portion of a bundle of the annealed and twisted hollow tube cord lengths with at least one closed end into a pot lifted off the bottom surface of the pot by approximately $1/8 - 1/4$ inch. Thermoplastic resin in pellet form is placed around the outside of the shell of the device, and the pot is heated to $275 - 277^{\circ}\text{C}$ for hollow fiber device. The resin melts and flows into the shell and between the lumens by head pressure and capillary action. An alternative method is to make a An external heating block is used for potting one end at a time. The perfluorinated thermoplastic end seals are preferably made of poly (tetrafluoroethylene-co-perfluoro (alkylvinylether)) having a melting point of from about 245°C . to about 260°C . A preferred potting material is Hyflon.RTM. 940 AX resin, from Ausimont USA Inc. Thorofare, N.J (now Solvay). Low viscosity poly (tetrafluoroethylene-co-hexafluoroprop- ylene) with low end-of-melt temperatures as described in U.S. Pat. No. 5,266,639 is also suitable. The process The method comprises vertically placing a portion of a bundle of the annealed and twisted hollow tube cord lengths with

at least one closed end into a pot lifted off the bottom surface of said pot by approximately $1/8 - 1/4$ inch. Thermoplastic resin in pellet form is placed around the outside of the shell of the device, and the pot is heated to 275 – 277 degree C for hollow fiber device. The resin melts and flows into the shell and between the lumens by head pressure and capillary action. A secondary process involves heating the potting material in a heating cup at around 275.degree. C. until the melt turns clear and are free of trapped bubbles. A recess is made in the molten pool of potting material that remains as a recess for a time sufficient to position and fix the fiber bundle and housing with the channels in its inner surface in place. Subsequently, the recess between fibers and channels in the housing will fill with the molten thermoplastic in a gravity driven flow. After cooling the potting process may be repeated for the second end of the device. After the potted ends are cooled, they are then cut and the lumen of the hollow tubes exposed. The potted surfaces are then polished further using a heat gun to melt away any smeared or rough potted surfaces. For module with a large number of hollow fiber, such as 2000 or more, it is possible that the module may potting defects which can be repaired using a clean soldering iron to fuse and close the damaged areas, or by melting new resin into the defect with the aid of a soldering iron.

[0028] A hollow fiber porous membrane is a tubular filament comprising an outer diameter, an inner diameter, with a porous wall thickness between them. The inner diameter defines the hollow portion of the fiber and is used to carry one of the fluids. For what is termed tube side contacting, the liquid phase flows through the hollow portion, sometimes called the lumen, and is maintained separate from the gas phase, which surrounds the fiber. In shell side contacting, the liquid phase surrounds the outer diameter and surface of the fibers and the gas phase flows through the lumen.

[0029] The outer or inner surface of a hollow fiber membrane can be skinned or unskinned. A skin is a thin dense surface layer integral with the substructure of the membrane. In skinned membranes, the major portion of resistance to flow through the membrane resides in the thin skin. The surface skin may contain pores leading to the continuous porous structure of the substructure, or may be a non-porous integral film-like surface. In porous skinned membranes, permeation occurs primarily by connective flow through the pores. Asymmetric refers to the uniformity of the pore size across the thickness of the membrane; for hollow fibers, this is the porous wall of the fiber. Asymmetric membranes have a structure in which the pore size is a function of location through the cross-section, section, typically, gradually increasing in size in traversing from one surface to the opposing surface. Another manner of defining asymmetry is the ratio of pore sizes on one surface to those on the opposite surface.

[0030] Manufacturers produce membranes from a variety of materials, the most general class being synthetic polymers. An important class of synthetic polymers are thermoplastic polymers, which can be flowed and molded when heated and recover their original solid properties when cooled. As the conditions of the application to which the membrane is being used become more severe, the materials that can be used becomes limited. For example, the organic solvent-based solutions used for wafer coating in the microelectronics industry will dissolve or swell and weaken most common polymeric membranes or thin walled hollow tubes. The high temperature stripping baths in the same industry consist of highly acid and oxidative compounds, which will destroy membranes and thin walled hollow tubes made of common polymers. High temperatures and pressures will deform and weaken many polymeric membranes and thin walled hollow tubes. Perfluorinated thermoplastic polymers such as poly(tetrafluoroethylene-co-perfluoro (alkylvinylether)) (poly(PTFE-CO-PFVAE)) or

poly(tetrafluoroethylene-co-hexafluoropropylene) (FEP) are not adversely affected by severe conditions of use, so that membranes and hollow tubes made from these polymers would have a decided advantage over less chemically and thermally stable polymers. These thermoplastic polymers have advantages over poly(tetrafluoroethylene) (PTFE), which is not a thermoplastic, in that they can be molded or shaped in standard type processes, such as extrusion. A modified PTFE, PTFM is suitable as a shell material or containment material for the lumens as this material is capable of bonding and adhering to the potted material, but is still not melt processable. Grooves, or channels in this material will have a superior characteristic over non grooved devices and the grooves serve as a sealing material, eliminate the need for secondary seals such as but limited to Kalrez, Viton, Chemraz. Perfluorinated thermoplastic hollow fiber membranes can be produced at smaller diameters than possible with PTFE. Fibers with smaller diameters, for example, in the range of about 350 micron outer diameter to about 1450 micron outer diameter, can be fabricated into contactors having high membrane surface area to contactor volume ratios. This attribute is useful for producing compact equipment, which are useful in applications where space is at a premium, such as in semiconductor manufacturing plants.

[0031] Being chemically inert, the Poly (PTFE-CO-PFVAE) and FEP polymers are difficult to form into membranes using typical solution casting methods as they are difficult to dissolve in the normal solvents. They can be made into membranes using the Thermally Induced Phase Separation (TIPS) process. In one example of the TIPS process a polymer and organic liquid are mixed and heated in an extruder to a temperature at which the polymer dissolves. A membrane is shaped by extrusion through an extrusion die, and the extruded membrane is cooled to form a gel. During cooling the polymer solution temperature is reduced to below the upper critical solution temperature. This is the temperature at or below which two phases form from

the homogeneous heated solution, one phase primarily polymer, the other primarily solvent. If done properly, the solvent rich phase forms a continuous interconnecting porosity. The solvent rich phase is then extracted and the membrane dried.

[0032] Preferred polymers are perfluorinated thermoplastic polymers, more specifically poly (tetrafluoroethylene-co-perfluoro (alkylvinylether)) (poly (PTFE-CO-PFVAE)), poly (tetrafluoroethylene-co-hexafluoropropylene) (FEP), or blends of these polymers, which are dissolved in a solvent to give a solution having an upper critical solution temperature, and which when the solution is cooled, separates into two phases by liquid-liquid phase separation. Teflon® PFA is an example of a poly (tetrafluoroethylene-co-perfluoro (alkylvinylether)) in which the alkyl is primarily or completely the propyl group. FEP Teflon® is an example of poly (tetrafluoroethylene-co-hexafluoropropylene). Both are manufactured by DuPont, Wilmington, Delaware. Neoflon™ PFA (Daikin Industries) is a polymer similar to DuPont's PFA Teflon®. A poly (tetrafluoroethylene-co-perfluoro (alkylvinylether)) polymer in which the alkyl group is primarily methyl is described in U.S. Pat. No. 5,463,006. A preferred polymer is Hyflon®. POLY (PTFE-CO-PFVAE) 620, obtainable from Ausimont USA, Inc., Thorofare, N.J. Thin walled hollow tubes such as preformed MFA Haflon® and FEP Teflon® tubes are available from Zeus Industrial Products Inc. Orangeburg, SC. Other thermoplastics or their blends which are useful in the practice of this invention include but not limited to poly(chlorotrifluoroethylene vinylidene fluoride), and polyvinylchloride, PES, UPE, HDPE, and other Polyolefins. The invention can also be used for similar but non identical materials such as PE potting material in a PP shell. Hollow thermoplastic tubes can be impregnated with thermally conductive powders or fibers to increase their thermal conductance. Examples of useful thermally conductive materials include but are not limited to glass fibers, metal nitride fibers, silicon and metal carbide fibers, or

graphite. The thermal conductivity of the hollow thermoplastic tubes or impregnated thermoplastic hollow tubes useful in this invention for energy exchange is preferably greater than about 0.05 watts per meter per degree Kelvin.

[0033] In another embodiment the hollow membranes or hollow tubes may be braided or twisted and optionally thermally annealed in a first step and then the individual tubes separated from each other after cooling to form self supporting helical shaped or non-circumferential shaped single tubes. Thermal annealing sets the crests and bends of the hollow tube so that the individual hollow tubes or cords can be separated and handled without straightening. These shaped hollow tube may be potted in the thermoplastic resin as described *vide supra*.

[0034] The term unified terminal end block or unitary end structure in the practice of this invention is meant to describe a mass or well of a thermoplastic resin with which one or more hollow tubes, hollow fibers, or cords of these have been mechanically bonded, chemically bonded, welded, or fusion bonded. Figure 3 illustrates an example of hollow tubes fusion bonded to a thermoplastic resin and filling a portion of one or more channels in the housing inner surface to form a unified terminal end block structure. The thermoplastic resin occupying a portion of the grooves and forming a unitary end structure with the grooves and housing walls may also be fuse to a layer of sintered thermoplastic material on the housing and groove surfaces. Optionally thermoplastic end caps having fluid connector ports or a thermoplastic housing may be fusion bonded to the one or more of the unified terminal end blocks as shown in FIG. 1.

[0035] The housing to form a single entity consisting solely of perfluorinated thermoplastic materials is prepared by preferably first pretreating the surfaces of both ends of the housing before the potting and bonding step. This is accomplished by melt-bonding or sintering

a powdered form of the thermoplastic potting material to the housing. The internal surfaces on both ends of the housing may be heated close to their melting point or just at the melting point and immediately immersed into a cup containing powdered [Polytetrafluoroethylene-co-perfluoromethylvinylether] thermoplastic potting resin available from Ausimont USA Inc. Thorofare, NJ. Since the surface temperature of the housing is higher than the melting point of the potting resins, the potting resin is then fused or sintered to the thermoplastic housing and any channels, grooves, or raised features for bonding the potting resin to the housing. The housing may be polished with a second heat step to fuse any excess un-melted thermoplastic powder. It is preferred that each end of the tube be treated at least twice with this pre-treatment.

[0036] The packing density of the hollow fibers, hollow tubes, cords and combinations of these within the shell tube should be in the range of from 3-99 percent by volume, and more preferably 20-60 percent by volume.

[0037] Hydrophobic microporous membranes are commonly used for contactor applications with an aqueous solution that does not wet the membrane. The solution flows on one side of the membrane and a gas mixture preferably at a lower pressure than the solution flows on the other. Pressures on each side of the membrane are maintained so that the liquid pressure does not overcome the critical pressure of the membrane, and so that the gas does not bubble into the liquid. Critical pressure, the pressure at which the solution will intrude into the pores, depends directly on the material used to make the membrane, inversely on the pore size of the membrane, and directly on the surface tension of the liquid in contact with the gas phase. Hollow fiber membranes are primarily used because of the ability to obtain a very high packing density with such devices. Packing density relates to the amount of useful membrane surface per volume of the device. It is related to the number of fibers that can be potted in a finished

contactor. Also, contactors may be operated with the feed contacting the inside or the outside surface, depending on which is more advantageous in the particular application. Typical applications for contacting membrane systems are to remove dissolved gases from liquids, "degassing"; or to add a gaseous substance to a liquid. For example, ozone can be added to very pure water to wash semiconductor wafers.

[0038] The contactor is comprised of a bundle of substantially parallel hollow fiber membranes potted at both ends and having unitary end structure(s) with the housing containing the fibers. The perfluorinated thermoplastic hollow fiber membranes of this invention are made of polymers such as poly (tetrafluoroethylene-co-perfluoro (alkylvinylether)), poly (tetrafluoroethylene-co-hexafluoropropylene), or blends thereof. The contactor has fluid inlets and outlet connections for the two fluids to be contacted. As illustrated in FIG. 1, fluid enters the contactor or exchanger through the fiber or tube lumens through fluid inlet connector, traverses the interior of the contactor while in the lumens, where it is separated from shell side fluid by the membrane or tube walls, the fluid exits the contactor or exchanger through the fiber lumens at fluid outlet connection. Shell side fluid enters the housing through a fluid fitting connection and substantially fills the space between the inner wall of the housing and the outer diameters of the fibers or tube, and exits through a second fluid fitting or connector. Preferably the fluid and the shell side fluid composition flow counter current to each other as illustrated in FIG. 1.

[0039] Porous or skinned hollow fiber diameters can range from 100-1000 μm in diameter. Wall thickness should be minimized and preferred thickness is 25-350 μm . Hollow fiber beds can consist of mats of fibers with thickness ranging from 1-25 cm in depth and length and width of 10-100 cm. The beds can be circular with diameters of 1-25 cm and lengths of 20-300 cm and contain multiple baffles to distribute gas throughout the bed of fibers. Hollow fibers

in the contactor may be straight or can be loosely packed. The hollow fibers may be extremely long and wrapped to a length nearly equivalent to the length of the device, effectively closing off the ends of the fiber to melt resin flow during the potting process.

[0040] Hollow tubes made from thermoplastics with outside diameters ranging from 0.007 to 0.5 inches, and more preferably 0.025 to 0.1 inches in diameter. For thin walled heat exchangers, preferably the hollow tubes may have a wall thickness ranging from 0.001 to 0.1 inches, preferably 0.003 to 0.05 inches in thickness. The hollow tubes can be used individually, or the tubes can be combined by braiding, plaiting, or twisting them to form cords comprised of multiple hollow tubes. The hollow tubes may be extremely long and wrapped to a length nearly equivalent to the length of the device, effectively closing off the ends of the fiber to melt resin flow during the potting process

[0041] In the practice of various embodiments of the present invention, combinations of porous and non-porous hollow tubes may be potted together. Such devices may be used to limit the amount of mass transferred while maximizing the amount of energy transferred between a process and an exchange fluid. For example temperature conditioned aqueous sulfuric acid may be re-circulated on the shell side of an exchange device to temperature condition air in a cleanroom and remove trace amounts of organic amines from the air. Temperature conditioning of the air is maximized by the number of non-porous potted hollow tubes while the amount of air in contact with the aqueous sulfuric acid scrubbing solution for mass exchange is controlled by the number of porous potted fibers present in the device.

[0042] Baffles may be useful in the practice of the present invention for enhancing the mixing and distribution of fluids on either side of the hollow tube contactor or exchange device (not shown in FIG. 1). The hollow fiber contactor or exchanger can be used in a single pass

mode or in a re-circulating mode for either or both the process fluid and or the exchange fluid. Preferably the contactor is provided with two or more fluid ports or fittings on the shell and lumen side of the housing. Usually one port serves as a fluid inlet and the second serves as a fluid outlet. The ports or fluid connections on the shell side of the contactor are separated from the lumen side inlet and outlet port by the fiber membrane or wall of the hollow tube. Preferably the fluid flowing within the tubes or and or fibers and the shell side fluid flowing on the outside of the tubes and or fibers flow counter current to each other as illustrated in FIG. 1; preferably the fluid flows in a manner which maximizes a cross flow of the fluids with respect to one another.

[0043] By fluidly sealed for purposes of the specification is meant that the potting resin has either fused with the thermoplastic tube or hollow fiber or has formed a mechanical bond with the fiber by entering and wetting the pores of the fiber with the thermoplastic. The seal is characterized in that fluid does not flow past the fibers or tubes in the potted area, rather fluid flows through inside of the membrane, in the case of porous fiber, or is confined to the inside of the hollow tubing and is physically separated from fluid on the outside of the hollow tubing or fiber.

[0044] Potting is a process of forming a tube sheet having liquid tight seals around each fiber. The tube sheet or pot separates the interior of the housing for the exchanger or contactor from the environment. The potting material is bonded to the housing including the channels or groove surfaces. This bonding may include physical mixing of melted materials as during welding or fusion of thermoplastics, mechanical interlocking of material, as well as chemical bonding of the materials. Preferably the bond between the housing and its grooves provides a fluid tight seal. The bond to form the unitary end structure may be formed by chemical reaction

between the potting material and the housing surfaces and housing groove surfaces. The bond may be the result of a union of the potting and housing materials by fusion, melting, or welding. Preferably the potting and housing, including any of the housing's coated surfaces, are thermoplastics that can be fused or welded together by various heating methods such as but not limited to welding, induction heating, ultrasonic bonding, infrared heating, and potting. The housing and potting material may be the same or different materials, for example the housing may be PFA and the potting MFA. The potting material can be thermally bonded to the housing vessel and channels, grooves, or raised structures on the inside of the housing in the present invention to produce a unitary end structure. The inside of the housing and channels may be coated with a layer of the potting resin sintered to the one or more inner housing surfaces to facilitate the bonding between the potting and housing. The unitary end structure comprises the portion of the fiber bundle which is encompassed in a potted end, the pot and the end portion of the perfluorinated thermoplastic housing, the inner surface of which is congruent with the pot and bonded to it. By forming a unitary structure, a more robust heat exchanger or mass transfer contactor is produced, less likely to leak or otherwise fail at the interface of the pot and the housing. The potting and bonding process is an adaptation of the method described in U.S. patent application Ser. No. 60/117,853 filed Jan. 29, 1999, the disclosure of which is incorporated by reference.

[0045] FIG. 2 illustrates different types of grooves- those in the potted region (G2, G3, G4) and those outside the potted region (G1). Grooves in the potted region are filled with the potting resin or thermoplastic. As shown in FIG. 2 B, a groove (G1) is added outside of the potted region. There may be one or more of these grooves outside the potted region and these grooves or channels may be on the inside or outside surface of the housing. Grooves including

but not limited to G2, G3, G4 are filled by potting resin and are considered to be in the potted region and form a unitary structure with the potting resin. The number of grooves and their surface area may be changed without limitation. The groove (G1) serves to reduce radial pressure on the pot – shell interface by hinging at this point. Without wishing to be bound by theory, the shell may be pressurized by the process fluid which tends to expand the thermoplastic shell. The stress relief on the inside diameter or the outside diameter will allow the shell to flex about this feature, thereby reducing stress on the pot- shell interface and maintaining its integrity.

[0046] As shown in FIG. 2, the grooves or equivalently channels in the housing may be made by machining or the housing may be molded with such grooves. Without limitation the grooves may be concentric and separated by equal or unequal spacing as shown in FIG. 2B; the grooves may be in the form of a spiral along the inside of the tube, they may consist of a series of groove channels along the axis of the housing; a hatched pattern, or a combination of these. The grooves or channels should be at least as deep as the potting material from the end of the housing, and the potting material may cover over the channels and extend into the housing. The channels and the housing wall may be covered or coated with a sintered thermoplastic material applied to the grooves as disclosed herein for bonding with the potting material. Preferably the depth of the grooves or channels permits the housing to maintain a pressure and temperature rating suitable for its use. One skilled in the art would know to look to ASTM tables to find the permitted wall thickness for the use of the potted device. Preferably the depth of the grooves or channels are less than about one half the thickness of the housing wall.

[0047] The grooves or channels may have a shape that maximizes surface area of contact and bonding between the potting material and the housing grooves. The depth and angles of the

sidewalls of the grooves may be made to vary the amount of bonding surface between the potting material and the grooves. Where an increase in the amount of shear component for the bond between the channel and potting is desired, deep thin channels are preferred. The additional surface area of the channels, some of which may not be parallel to the housing walls, result in fusion and adhesion of the potting resin to all faces or surfaces of the groove. The radial force created by thermal or pressure expansion of the device during use may have a portion of this force transferred to a shear component through bonding of the potting resin with the surfaces of the grooves which greatly improves the strength of the device.

[0048] While grooves and channels are preferred for bonding the thermoplastic resin to the housing of the present invention, it is also contemplated that raised structures permanently bonded or fused onto the inner surface of the housing tube could be made and used with the same effect as channels or grooves for purposes of bonding the thermoplastic potting resin to the housing. Such raised structures may be considered as an equivalent to grooves or channels for purposes of the present invention. Preferably the structures result in bonding or fusion between the raised structures and the potting resin. Preferably the bonding transfers a portion of the radial force into a shear component of force between the potting resin and the raised structure.

[0049] It is also contemplated that additional means for reducing stress between the housing and the potting material may be used in addition to channels or grooves in the housing. For example a housing with channels in the inside of the housing may have the outer wall of the housing thinned by machining to relieve pressure on the interface between the potting material and the shell. The thinned material will yield to material movement more readily, allowing temperature and pressure effects to be self compensating by flexible components to maintain the integrity of the bond between the potting resin fused to the housing.

[0050] Various aspects of the present invention will be illustrated with reference to the following non-limiting examples.

EXAMPLE 1

[0051] This example compares the ability of various potted devices to withstand stress testing.

[0052] Below is a table showing the advantage of adding the grooved interface. The original PFA design showed loss of housing to potting material integrity at 120°C. The temperature/ Pressure test is a method of accelerating long term ambient conditions for the device. The MFA only device showed loss of bond integrity at 150°C. All tests on MFA devices with the improved interface are integral up to 200°C and beyond, the PFA device with the improved groove or channel interface is also integral at temperatures up to 160°C. This device was destructively tested after the 160°C to determine the strength of the bond. The strength of the bond is determined by cutting off thinning the outside of the device leaving approximately .080 - .100" wall thickness. Axial and circumferential cuts are made into the shell approximately .25" above the potted area, leaving a tab approximately .5" wide by .25 long. The tab can then be used to pull up on the material in an attempt to pull apart the shell material from the potted material. In this manner the strength of the bond can be tested qualitatively by force, or quantitatively by an instrument such as an Instron.

[0053] This process has not shown to improve the adhesion of the potted material to the shell, only to improve the overall strength of the device by eliminating stress at this interface, and transferring the stress to the potted material entrapped in the grooves. Without wishing to be bound by theory, the grooves, and their additional surface area, some of which is not parallel to the housing walls, also add adhesion of the potting resin to at least a portion, and preferably all

surfaces or faces of the groove, adding a shear component to a radial force created by thermal or pressure expansion of the housing shell. This shear component greatly improves the strength of the device.

TABLE 1

Test Condition	120°C, 70psig,5h	130°C, 70psig,5h	140°C, 70psig,5h	150°C, 70psig,5h	*160°C , 5hrs	*170°C , 5hrs	*180°C , 5hrs
PFA tube, original	integrity loss						
PFA tube w/stress groove relief	integrity loss						
PFA tube w/grooved ID	Passed	Passed	Passed	Passed	Pass		
MFA, original	Passed	Passed	Passed	integrity loss			
MFA tube w/stress groove relief	Passed	Passed	Passed	Passed	Pass	Pass	Ongoing
MFA tube w/grooved ID	Passed	Passed	Passed	Passed	Pass	Pass	Ongoing
Tubes flamed with rotational fixture, MFA original		Passed	Passed	Ongoing			
Tubes flamed with rotational fixture, MFA threaded ID		Passed	Passed	Ongoing			

EXAMPLE 2

[0054] The potted devices of the present invention may be used for heat and or mass exchange including but not limited to filtration, gas contacting, heat exchange, gas scrubbing and combinations of these. The potted devices may be placed in an apparatus for cleaning or chemical modification of substrate surfaces including but not limited to single wafer cleaning tools, re-circulating cleaning baths, temperature conditioning of fluids prior to disposal (such as

hot sulfuric acid used to remove polymer coatings from optical fibers and photoresists from coated silicon wafers). As illustrated in FIG. 4 an exchange fluid in a tank, a chiller, or a water supply loop is flowed through one side of the potted heat exchanger. A pump may be included to re-circulate the exchange fluid if necessary and particle filters and valves may also be used. Fluid from the a cleaning or process bath, including but not limited to acids, bases, and oxidizers, may be re-circulated on the other side of the exchange device and its temperature conditioned by heating or cooling it. The temperature conditioned fluid is returned to the process bath or tool for use on the substrates. As shown in FIG. 5, a potted exchange device of the present invention may be used to cool a process or cleaning fluid prior to discharge. An example of such fluids includes but are not limited hot sulfuric or phosphoric acids.

[0055] The present invention may also relate to a method and apparatus for the purification of gases which may be used in chemical processes or which may be removed from an effluent stream. In particular, the present invention provides a device which maintains the integrity of the potting resin and housing seal during exothermic scrubbing reactions where for example an exhaust fluid is purified by reacting a component of the exhaust fluid with a reactive liquid, gel, or slurry contained on one side of a porous hollow fiber membrane potted into a housing having grooves, the potting forming a unitary end structure with the housing and fluidly sealing the hollow fibers potted in the resin.

[0056] Although the present invention has been described in considerable detail with reference to certain preferred embodiments thereof, other versions are possible. Therefore the spirit and scope of the appended claims should not be limited to the description and the preferred versions contain within this specification.

CLAIMS

What is claimed is:

1. An exchange device comprising:

a thermoplastic housing having one or more hollow tubes; said hollow tubes fluidly sealed by a thermoplastic resin and said resin fluidly sealed (by fusion) to an end portion of said housing by one or more grooves in said housing; said grooves and resin forming a unitary end structure wherein said resin and housing fuse at a portion of said groove to form said unitary end seal.
2. The exchange device of claim 1 having a sintered thermoplastic coating on the inside of said housing.
3. The exchange device of claim 1 wherein said housing includes fluid fittings.
4. The exchange device of claim 1 wherein said thermoplastic includes MFA.
5. The exchange device of claim 1 wherein said hollow tube are porous hollow fibers, skinned hollow fibers, thermoplastic tubes, or combinations of these.
6. The exchange device of claim 1 wherein the ends of the hollow tubes are open.
7. An exchange device comprising:

a thermoplastic housing having one or more fluidly sealed hollow tubes in a thermoplastic resin; said resin occupying a volume of one or more grooves on an interior surface of said housing; said grooves and resin forming a unitary end structure wherein said resin and housing fuse at a portion of said groove to form said unitary end seal.

8. An apparatus for exchanging energy with a fluid comprising:

the exchange device of claim 1 having a fluid inlet and outlet connected for receiving and delivering a fluid into a re-circulation loop ; said exchange device having an exchange fluid inlet and outlet fittings for flow of an exchange fluid; said exchange fluid separated from the treated fluid by the hollow tubes and exchanging energy with said fluid;

a re-circulating pump, said pump in fluid communication with the fluid inlet exchange device; and

a tank for holding an article to be treated by said fluid and including a volume of fluid , said tank

9. The apparatus of claim 8 further comprising a particle filter.

10. The apparatus of claim 8 wherein the exchange fluid includes water.

11. The apparatus of claim 8 wherein the article to be treated includes silicon

12. An exchange device comprising:

a thermoplastic housing having one or more fluidly sealed hollow tubes in a thermoplastic resin; said resin occupying a volume of one or more grooves on an interior surface of said housing; said grooves and resin forming a unitary end structure wherein said resin and housing fuse at a portion of said groove to form said unitary end seal; and

a fluid at a temperature of at least 60 °C

13. The device of claim 12 wherein the pressure of the fluid is at least 10 psig.

14. A method of making an exchange device comprising:

flowing a thermoplastic material into a housing with grooves and including one or more hollow tubes; and

forming a fluid tight seal between said thermoplastic material and said hollow tubes and a fluid tight seal between said thermoplastic and said housing; said thermoplastic occupying a portion of said grooves and fusing with a portion of said housing.

15. The method of claim 14 wherein said housing has a coating of a sintered thermoplastic material on one or more of housing surfaces.

16. A method of treating a fluid comprising:

flowing a fluid to be treated on a first side of at least one hollow tube having two sides and a thermoplastic wall interposed between them, said tube potted in a fluid tight manner within a thermoplastic material, said thermoplastic material bonded to a housing with grooves, at least a portion of said grooves filled with and bonded to the thermoplastic material, forming a fluid tight seal between said thermoplastic material and said housing; and

exchanging energy or mass with said fluid to be treated by flowing an exchange fluid on a second side of said hollow tubes, the energy or mass being transferred through the hollow tube wall.

17. The method of claim 16 wherein said tube wall is porous.

18. The method of claim 16 wherein thermal energy is transferred.

19. The method of claim 16 wherein said tube wall is non-porous.

20. An exchange device comprising:

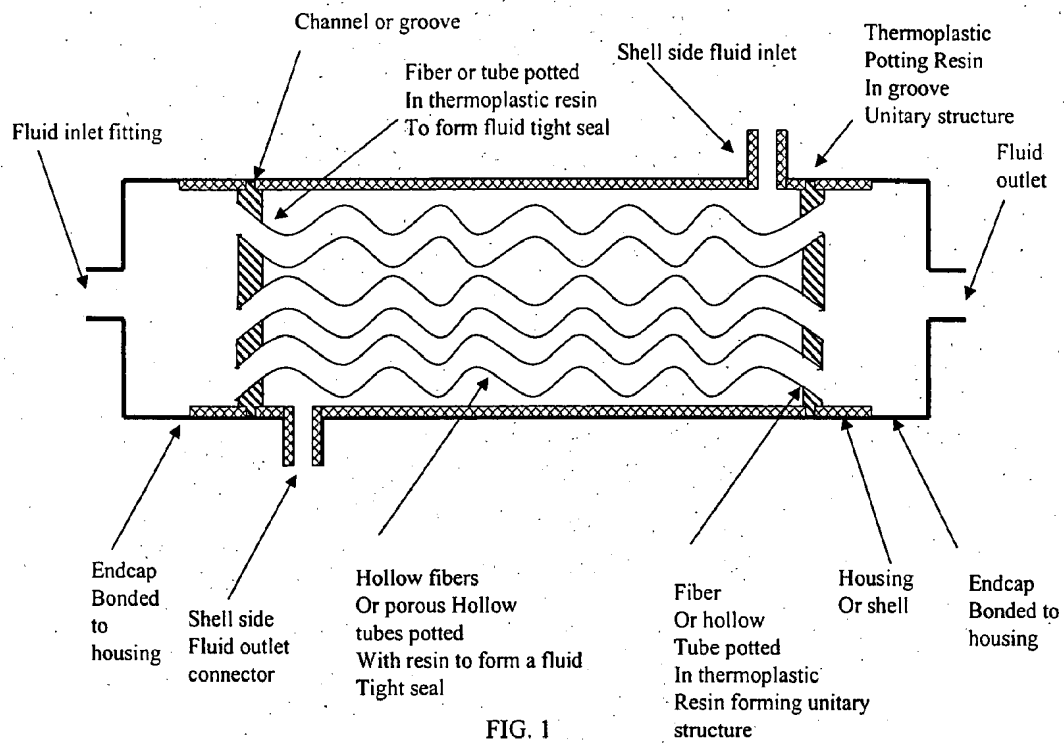
a thermoplastic housing having one or more hollow tubes; said hollow tubes fluidly sealed by a bond with a thermoplastic resin and said resin fluidly sealed by a bond to an end portion of said housing by one or more grooves in said housing; said grooves and resin forming a unitary end structure wherein said resin and housing bond at a portion of said groove to form said unitary end seal.

21. An exchange device comprising:

a thermoplastic housing having one or more hollow tubes; said hollow tubes fluidly sealed by a bond with a thermoplastic resin and said resin fluidly sealed by a bond to an end portion of said housing by one or more grooves in said housing; said grooves and resin forming a unitary end structure wherein said resin and housing bond at a portion of said groove to form said unitary end seal and add adhesion of the potting resin to one or more surfaces or faces of the groove, adding a shear component to a radial force created by thermal or pressure expansion of the housing shell.

ABSTRACT OF THE DISCLOSURE

The present invention relates to potted exchange devices including hollow tubes and or hollow porous membranes wherein the housing includes recessed channels or grooves. The recessed channels or grooves are filled by the potting material during the potting process and form a unitary end structure with the potting material. The grooves or channels formed on the inside of the housing maintains the integrity of the potting with the housing under a wide range of mechanical and thermal conditions.



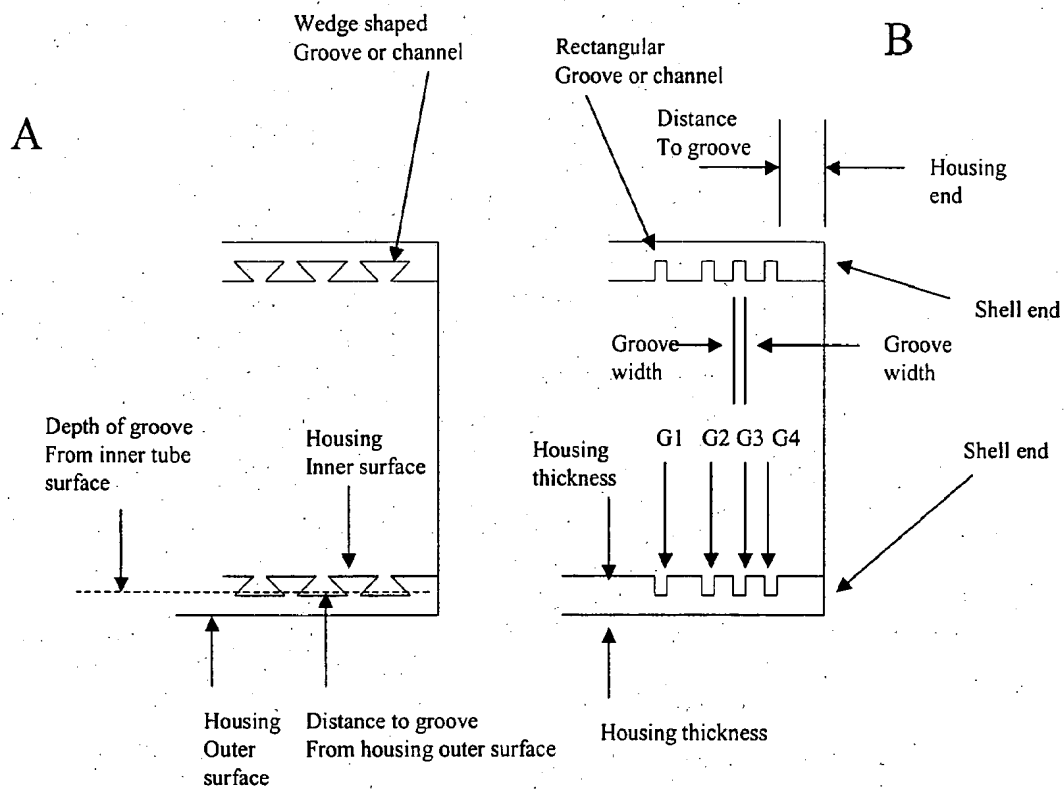


FIG. 2

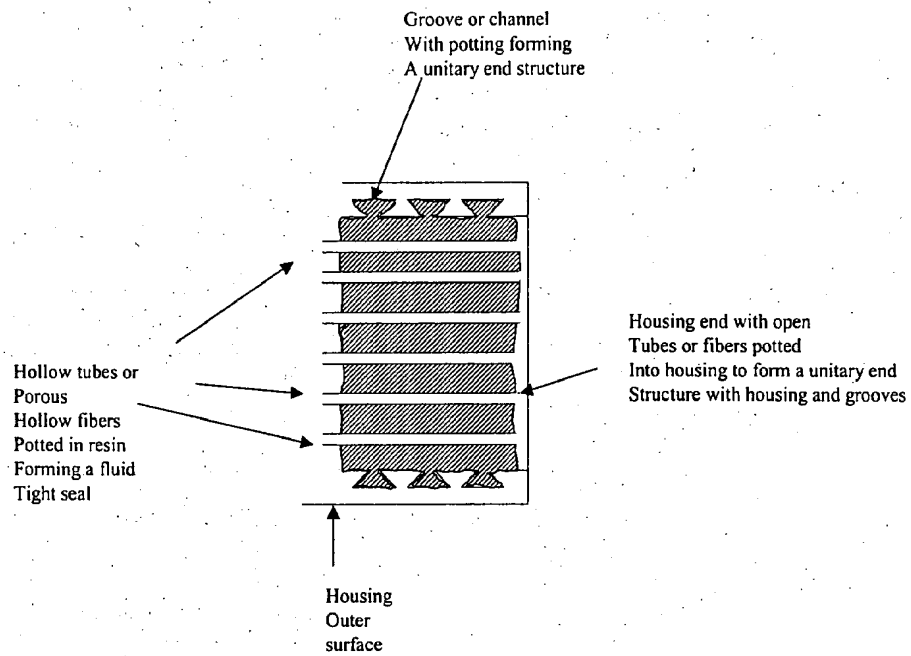


FIG. 3

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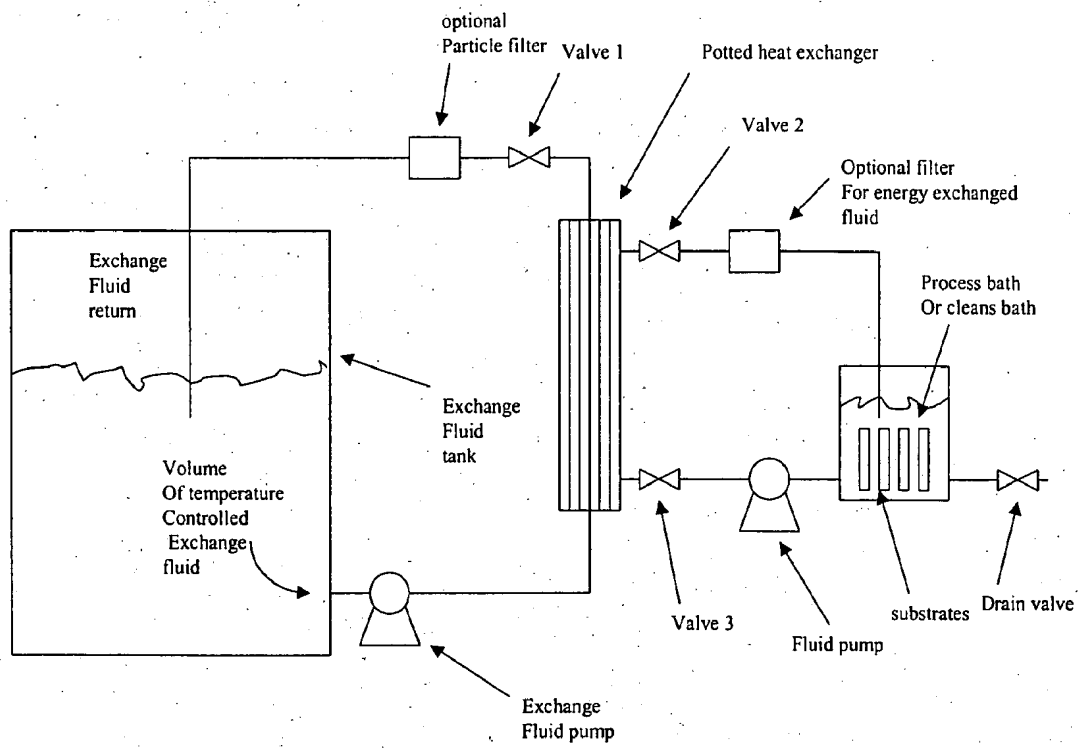


FIG. 4

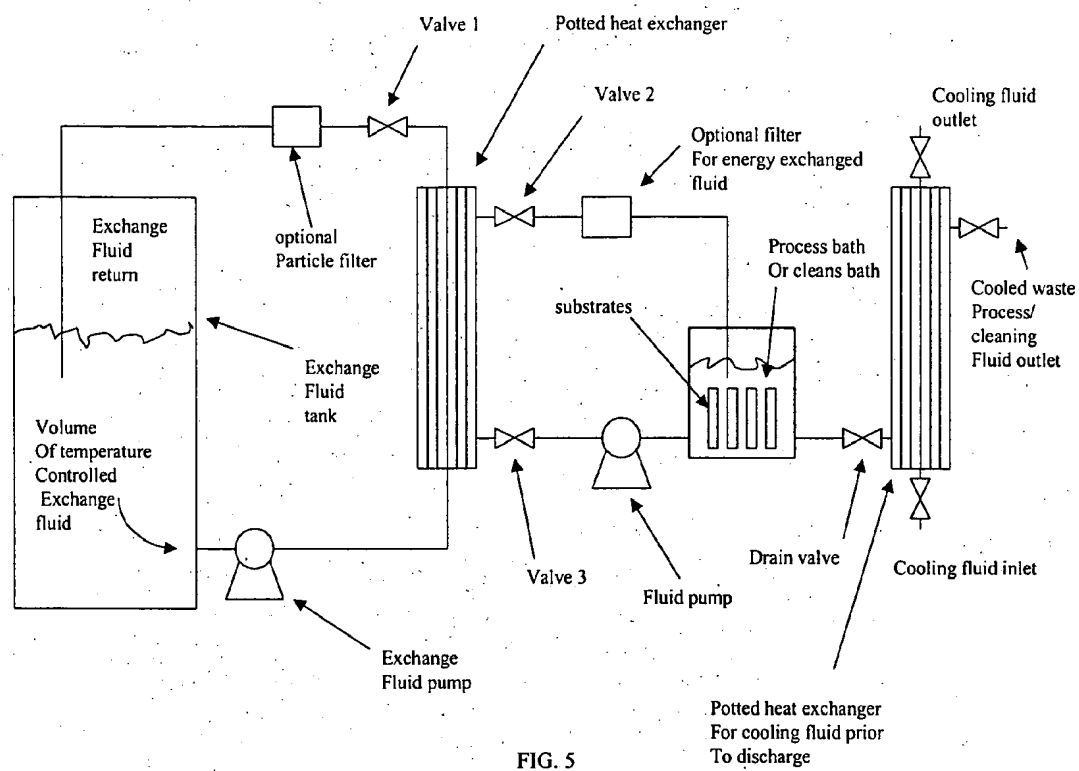


FIG. 5

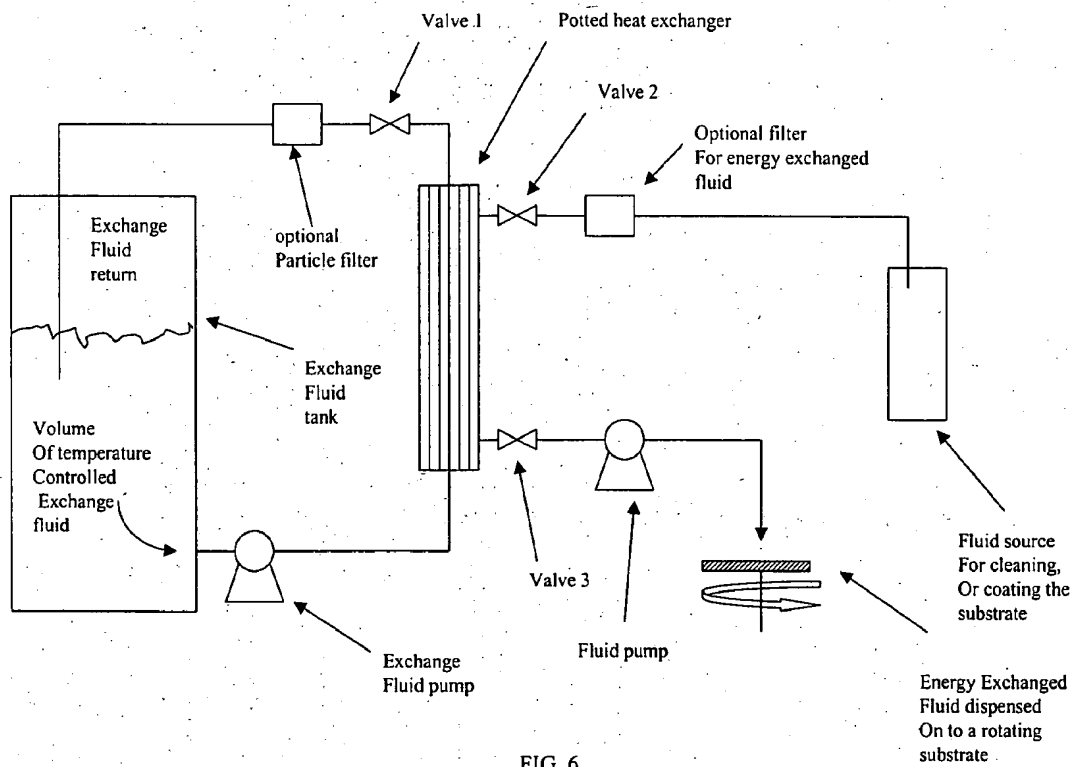


FIG. 6